

STANDARD OPERATING PROCEDURE

TITLE: Total Solids Determination

DEPARTMENT: Receiving/Metals Prep

SCOPE: The purpose of this SOP is to document the procedure for the network weighing operations for the initial and final weight. This applies to solids or sludges only.

REFERENCES: Standard Methods for the Examination of Water and Wastewater, 1992, 18th Edition, Method 2540B (modified)

APPLICATION: Percent solids are determined for compositional samples which are reported on a dry weight basis.

PROCEDURE SUMMARY:
A portion of the sample is transferred to a pre-weighed evaporating dish and evaporated to dryness at 103-105°C.

APPARATUS AND MATERIALS:

Aluminum weighing dishes
Analytical Balance, Mettler AE 160, 0.1 mg capacity, or similar balance.
Drying oven

PROCEDURES PERFORMED IN RECEIVING:

INITIAL WEIGHING

NOTE: Soil samples to be analyzed for volatiles which have only 1 sample jar submitted should not have TSCs performed on them at Receiving.

Write "needs TSCs" on the jar lid. The volatiles group will submit the sample to Receiving for a TSC analysis after the volatile analysis is complete.

- 1.0 Computerized operation:
 - 1.1 Zero the balance with the "Tare" button.
 - 1.2 Login on the computer and open the "WET WEIGHTS" program.
 - 1.3 Write the sample # of the sample to be weighed on the aluminum weighing dish bottom.
 - 1.4 At the "Enter Lab #" prompt, type in the sample # of the sample to be weighed.
 - 1.5 Place the aluminum weighing dish on the balance pan. When the "g" is solid (not flashing) on the balance, press enter on the keyboard.

- 1.6 The computer screen will read "add sample and press Enter." Add 5-10 grams of homogenized sample to the dish. When the "g" is solid (not flashing) on the balance, press enter on the keyboard.
- 1.7 Remove the dish with sample and place it on the oven tray.
- 1.8 Continue Steps 1.3 through 1.6 until the necessary number of samples have been weighed.
- 1.9 Exit the program by pressing enter.
- 1.10 A print out of the sample numbers and corresponding initial weights will automatically print out after "ending" the session on the computer. A print out may be generated at any time by pressing the print button on the balance.
- 1.11 Review the print out for sample #, the weights of the aluminum weighing dish (approximately 0.9 - 1.1 g) and the weight of the sample (5 - 10g). If there is a problem with a weight or the sample #, single line cross out the line in question.
 - 1.11.1 Redo the questionable sample.
 - 1.11.2 Give the original printout to the systems analyst, to delete the original suspect data. He/she will delete the error, initial it and return it to you.
- 1.12 Initial and date the print out. Place print out in sample receiving bin.
- 1.13 When finished weighing the samples, clean off the balance and surrounding area.
- 2.0 Transport the dishes, with samples, on the oven trays to the Wet Chemistry lab and place in the oven at 103° - 105° C. Document the time the samples go into the oven on the Time Sheet for TSC Oven (posted on oven).
- 3.0 QC
 - 3.1 One in twenty samples will be duplicated.
 - 3.2 Open the "DUPWET" Program.
 - 3.3 Weigh the duplicate sample, following steps 1.3 through 1.9.
 - 3.4 Record "DUP" next to the appropriate sample on the printout to indicate that the entry is for the duplicate. Staple "Dup" printout to wet weight printout.

The later half of this procedure will be completed by the Metals Prep group.

Collect wet weight sheets from sample entry. Make any changes or deletions needed.
Place sheets in wet weights binder.

PROCEDURES PERFORMED IN METALS PREP:

- 4.0 Dry the sample overnight (12-24 hours).

Quality Assurance Document

DRY WEIGHT REHEATS

- 4.1. To demonstrate the attainment of constant weight, data must be recorded for a minimum of two repetitive weigh/dry/desiccate/weigh cycles with a minimum of 1 hour drying time in each cycle, for 1 sample in each batch of 20. Constant weight would be defined as a loss of no greater than the annually calculated RPD control limit between the start weight and final weight of the last cycles. The demonstration of attainment of constant weight for the batch is to be recorded in a department daily log.

FINAL WEIGHING.

5.0 Computerized operation:

- 5.1 Open dry weight program by double-clicking Dry Weight icon.
- 5.2 At the "Enter Lab #" prompt, type in the sample # of the sample to be weighed. Hit Enter.
- 5.3 Place the sample on the scale and when the "g" is not flashing, hit Enter.
- 5.4 At the prompt, type "S" or "B" for appropriate matrix type. Hit Enter.
- 5.5 Continue steps 5.2 through 5.4 until the necessary number of samples have been weighed.
- 5.6 Exit the program by pressing enter.
- 5.7 A message will come up that asks if this is the last run of dry weights. If so, the file must be prepared for the export procedure. Type "Y" after the question "Ready to send to LIMS?" Now a file has been set up that can be sent to LIMS. The report will be sent to the printer. If report does not print, go to F:\Balance\Dryprt.prt print.
- 5.8 Double click on Duplicate Dry Weights icon.
- 5.9 Enter the appropriate lab numbers. Hit enter to exit the program. This report will be sent to the printer. If report does not print, go to F:\Balance\Dryprt.prt print.
- 5.10 The acceptability of the duplicate sample should be evaluated. The computer calculates the difference between the original sample and the duplicate. If the Relative Percent Difference (RPD) is greater than the annually calculated control limit, the duplicate will have to be reweighed, redried and recomputed.

Staple both printouts (% solids and duplicate) together.

Calculations

The following calculation has been automated:

$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

6.0 Import data into LIMS.

- 6.1 Sign onto Conifer.

- 6.2 Choose "Import Data".
- 6.3 Select "Browse" to find the data file that you just made.
- 6.4 For file type, choose: F:\Balance\Output.dat. Under "List Files of Type", select "**.*" and click on "OK".
- 6.5 You'll come to the screen titled "Import Data Options".
 - 6.5.1 Under "import file path & name", click on the field "Names in row 1".
 - 6.5.2 Click on "Start Import".
- 6.6 The next screen will be the "Import Data Screen". All sample #s and numeric results are there. Click on "Send to LIMS".
- 6.7 You'll come to the "Form: Import Worksheet" screen, containing existing worksheets.
 - 6.7.1 Click on "Create new WS".
 - 6.7.2 Choose "yes" to "Custom Data Entry Form".
 - 6.7.3 If the blue screen titled "Imported Data" comes back blank, proceed to step 6.7.4. If a red warning square appears with test IDs that did not go into LIMS, press the printer button and print the exception report.
 - 6.7.4 Click door to go back to main menu. Click on "Enter sample results".
 - 6.7.5 "Select a method for entering results" will appear. Click on "By worksheet".
 - 6.7.6 Highlight new worksheet, click on "Modify Description".
 - 6.7.7 Type in a worksheet description (dry weight-date) and hit continue twice.
 - 6.7.8 On this gray screen, hit "Mark Status". At the "Status Change" screen, click on "Mark tests analyzed". Hit "OK" to mark all tests in the worksheet. Put in the date under "analysis date" and hit "OK". Enter initials into LIMS. Exit the program by clicking on the door to go back to the main menu.

QUALITY CONTROL

Precision:

A duplicate must be analyzed on each group of samples of a similar matrix type at a frequency of the RPD determined by control limits calculated annually.

The RPD of the duplicate and the original sample must be less than 14%.

DATA DELIVERABLES/DOCUMENTATION:

All reports and documentation must be legible, single-sided, and clearly labeled.

Quality Assurance Document

Sample analysis reports will include: sample result, units, project name, laboratory name, station ID, laboratory sample number, sample collector, project number, collection date, report date, work order number, comments describing in detail any problems encountered in processing the sample, and signature of the sections supervisor.

The following documentation will be completed but not submitted as a deliverable unless requested: summary of QC sample analyses, raw data (copies of bench sheets), and laboratory log books (standard logs).

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

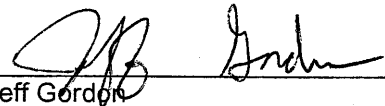
POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.


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